HIGH TEMPERATURE THERMOCOUPLE
RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 9
Period 1 February 1964 to 1 March 1964
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
GEORGE C. MARSHALL SPACE FLIGHT CENTER
Huntsville Alabama

work performed by
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ABSTRACT

This report covers the period 1 February 1964 to 1 March 1964, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C range, under adverse conditions of oxidation, erosion, vibration and shock.

The primary objectives of the program are to advance the state-of-the art of high temperature thermometry and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Three of the second generation gauges were delivered to N.A.S.A. on 26 February 1964. Calibrations of these gauges were made from about 1700°F to 5400°F.

A review of progress to date was held in conference with M-ASTR I personnel.

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SECTION I

SUMMARY

1 0 Period Covered

This report covers the period 1 February 1964 to 1 March 1964.

1 1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion oxidations and high stress levels for useful period of time Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle

To accomplish the above objectives the Contractor shall consider and explore specific R & D efforts as follows:

a. Development of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.

b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.

c. Determine the best combination of compensated lead wires for use with the immersion type probes.

d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.

1.1 Statement of Work (Contin)

e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output.

f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

a. Fabrication

Six of the second generation type 4735 gauges were fabricated during the current reporting period. Three of these gauges Serial Numbers 007, 008 and 009, were delivered to N.A.S.A. on 26 February 1964.

b. Calibrations

Calibrations were run on the second generation gauges in the ACL high temperature calibration oven, with a micro-optical pyrometer. The highest temperature at which a stabilized output was obtained was 5430°F, the upper limit of the Pyrometer.

c. Conferences

A discussion of the project to date was held at M-ASTR I. Major points discussed were calibrations and protective coatings.

SECTION II

PAST PROGRESS

2.0 General

Previous effort was reported in ACL Progress Reports T-1097-1 through T-1097-8.

2.1 Prototype Design and Development

As was previously reported, objectives for the first prototypes were limited to the 4000°F - 4500°F range in the interest of accumulating test data for analysis, the results to be utilized in future design.

A design approach for the prototype gauges was selected, and drawings prepared, detailing means of fabrication and assembly.

Investigations made into fabrication techniques involved in working vapor deposited Tungsten, resulted in improved material handling techniques.

Shock and vibration tests, performed on a prototype mock-up, resulted in a conclusion that the sheath material was intrinsically capable of withstanding the specified shock and vibration requirements.

Samples of various types of compensation lead wires were ordered for test and evaluation.

An evaluation of the SRI calibration tests for ACL Type 4734 gauges was made, resulting in a conclusion that an optimum immersion depth might be in the order of 1-1/2 inches in an isothermal region.

The two Type 4734 gauges tested by N.A.S.A., and returned to ACL were examined and results of the examination were reported.

[redacted]

2.1 Prototype Design and Development (Contin.)

A test of a "no-insulation" approach was started, but was aborted due to a failure in the test oven. Such tests were subsequently continued.

Three prototype gauges were delivered to M-ASTR-I, on 17 October 1963, for test and evaluation. Calibration of this type of gauge indicated a shift in emf output to a higher value than that shown in previous calibrations. The shift was believed due to a spurious emf contributed by the "compensated" lead wires. The curves, however, paralleled the curves taken by Southern Research Institute, as well as those predicted by ACL.

Further tests verified the presence of lead wire errors.

Analyses of form and shock drag loads were made. The results will be considered in future design.

Investigations of oxidation resistant coatings were continued. Accumulated data was reviewed, and tabulated for comparison and reference.

Response tests performed on one Type 4735 gauge yielded response as low as 45 milliseconds from ambient air to boiling water. Lead wire tests resulted in a conclusion that the thermocouple materials should be used in lead extensions for best accuracy. Further investigations of oxidation resistant coatings, and insulators verified the conclusion reached in earlier tests. Design of the second generation gauges was continued.

Three second generation gauges were delivered to M-ASTR-I on 26 February 1964 for test and evaluation. These gauges incorporated thermocouple materials as lead wire, and elimination of BEO insulation in the Hot Zone. Calibrations of this type of gauge showed an increase in the upper temperature limit, and virtual elimination of lead wire error. emf output curves essentially tracked predicted values.

SECTION III

CURRENT PROGRESS

3.0 General

Effort during the current reporting period was directed principally toward fabrication of the three second generation gauges scheduled for delivery during February 1964, calibrations in the 4000°F to 5000°F temperature range, and delivery of the three gauges.

3.1 Progress

3.1.1 Second Generation Gauges

Six of the Type 4735 Thermocouple sheath assemblies were fabricated during the report period. Four of these assemblies were assembled into the configuration described in report No. T-1097-8, two were held in reserve for future tests. Of the four assembled, three were delivered to N.A.S.A., the other was used for calibration and test.

3.1.2 Delivery

Three Type 4735, second generation gauges were delivered to M-ASTR-I on 26 February 1964. These gauges bore serial Numbers 007, 008, and 009.

3.1.3 Calibrations

Two gauges were available for calibration and test. Serial No. 003 was assembled with a sheath from the first run. The other, Serial No. 010, was assembled with a sheath from the second run. The principal difference between the two was a greater wall thickness in the second generation gauge sheaths. The principal interest in calibrating the two gauges under the same conditions was to verify whether it was possible to duplicate the output characteristics of the gauges between runs. Previous tests had demonstrated that the characteristics of gauges fabricated with sheaths from a single

[redacted]

3.1.3 Calibrations (Contin.)

run were very close to identical. However, the question of repeatibility had been raised by both ACL and M-ASTR-I and a determination was deemed advisable.

3.1.3.1 Test Method

The calibration oven described in the last report was used in all the calibrations reported herein. The micro-optical pyrometer was set up with a focal length, such that the standard tungsten lamp could be compared in brightness, with the thermocouple within the cavity and in proximity radially, with the thermocouple junction. Except as noted, the brightness comparisons were made only when the thermocouple tip had been scanned and there was no evidence of gradients. Since the tungsten thermocouple sheath was entirely enclosed within the tungsten cavity (with the exception of the .062 inch diameter sighting hole) it was assumed that no correction for emissivity need be made. The very good agreement with the Hoskins and Englehard curves for Tungsten-Tungsten 26% Rhenium Thermocouples at a large number of points seems to validify this assumption. Immersion depths were recorded for each run. Since the gauge sheaths are tapered, rather than cylindrical, the effective immersion depth is greater than would be the case with a cylindrical gauge, since the ratio of immersion depth to probe diameter would be calculated on the basis of the mean sheath diameter for the immersion used. This ratio is estimated at six, rather than four. Again, good agreement with the literature on the effect of immersion depths is evidenced in the calibration curves. The literature recommends that immersion ratios of six to ten be used.

The thermocouple output was referenced to 32°F (ice bath) in all runs. The output was measured with a Leeds & Northrup Type 8662 Precision Potentiometer.

The output curves for three immersion depths, .875 inch, 1.00 inch and 1.25 inches are shown in Figure 1.

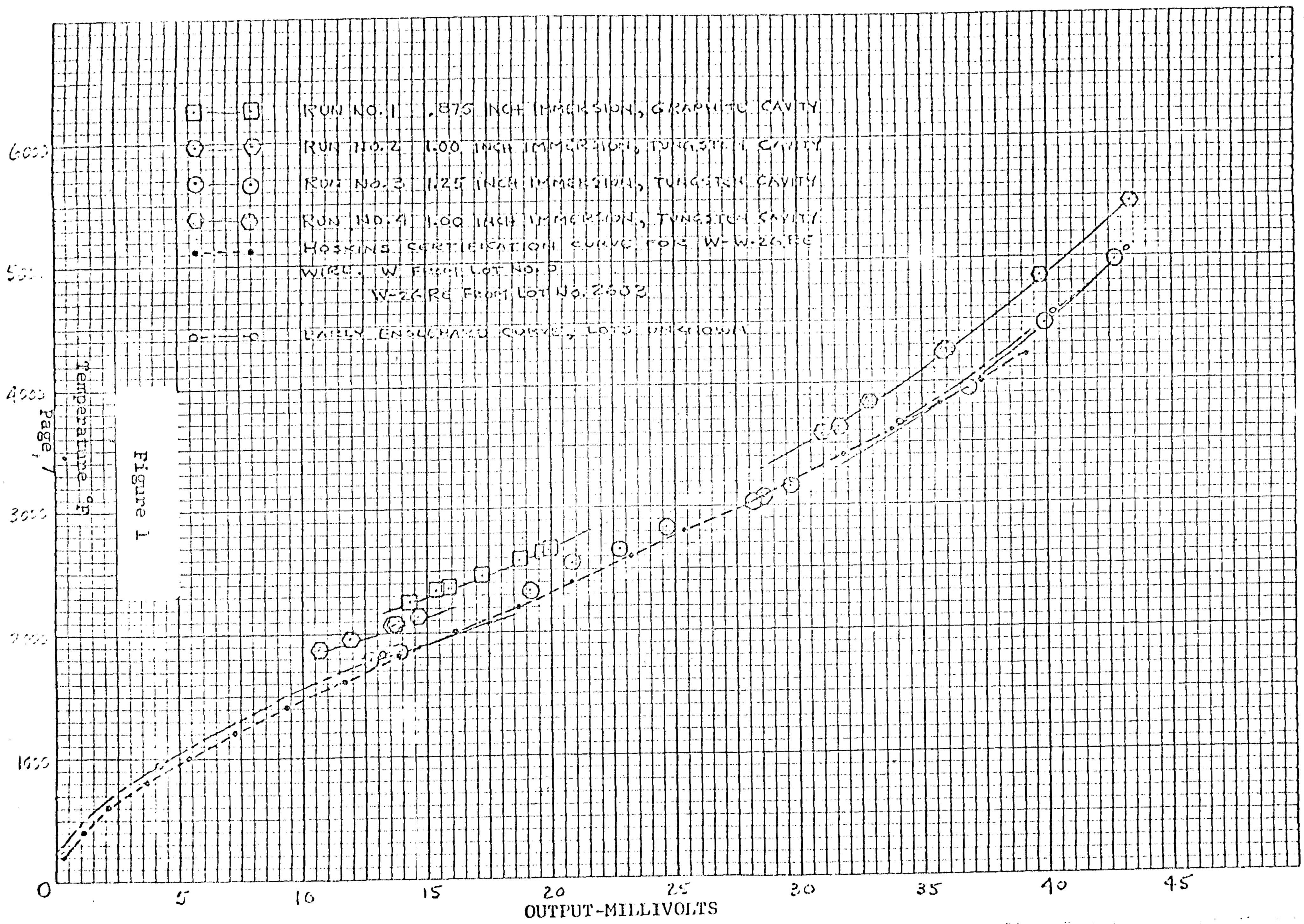
RUN NO. 1 .875 INCH IMMERSION, GRAPHITE CAVITY
RUN NO. 2 1.00 INCH IMMERSION, TUNGSTEN CAVITY
RUN NO. 3 1.25 INCH IMMERSION, TUNGSTEN CAVITY
RUN NO. 4 1.00 INCH IMMERSION, TUNGSTEN CAVITY
HOSKINS CERTIFICATION CURVE FOR W-W.26RE
WIRE: W FROM LOT NO. P
W-26RE FROM LOT NO. 2602
EARLY ENGELHARD CURVE, LOTS UNKNOWN
Temperature °F
6000
5000
4000
3000
2000
1000
0
5
10
15
20
25
30
35
40
45
OUTPUT-MILLIVOLTS

Figure 1

3.1.3.1 Test Method (Contin.)

The data points taken at .875 inch immersion during Run No. 1, were taken with the thermocouple enclosed in a graphite cavity. These were halted, however, and tests continued with the tungsten slug, in Runs 2, 3, and 4. The separation apparent between the curves for different immersion depths is in good agreement with data taken by SRI and ACL on earlier probes.

During the runs, the immersion depths were changed from 1.00 to 1.25 inches several times to verify the repeatibility of the two different gauges. Thus, the curves are representative of two different production runs.

A tabulation of the data taken during the runs is shown below in Table I. A tabulation of the Hoskins Certification for the wire is shown in Table II. It should be noted that the Hoskins curve is for their lot No. 5 Tungsten wire vs Lot No. 2603 Tungsten-26% Rhenium Alloy Wire. In the Type 4735 gauges, the Lot No. 2603 Tungsten Rhenium alloy was used, but the tungsten leg is made of thermochemically formed tungsten which is of much greater purity than normal tungsten wire. Therefore, some difference in the output characteristic could be expected.

The ACL calibration at 1.25 inch immersion shows a similar "hump" in the curve between 2000°F and 3000°F as does the Hoskins curve. The reason for this "hump" is not known. Data points were not taken in this range during Runs No. 2 and 4 at 1.00 inch immersion. However, it is likely that the curve would have taken the same shape. The early Englehard Curve is plotted in Figure 1 and, although differences are seen, it is of interest that agreement is, in general good.

[redacted]

TABLE I

CALIBRATION - TYPE 4735 GAUGE

(Ref. Junction - 32°F)

Run No.	Pyrometer °F	Output MV	Run No.	Pyrometer °F	Output MV
1	2264	14.47	3	3164	29.62
.875	2327	15.45		3937	36.90
Immersion	2352	15.95		4460	39.90
	2458	17.25		4975	42.70
	2592	18.75	4	3596	30.95
	2622	19.67	1.00"	3614	31.60
	2640	19.95	Immersion	3821	32.80
2	1886	10.68		4208	35.83
1.00"	1958	11.94		4289	35.90
Immersion	2052	13.55		4830	39.75
	2067	13.66		5430	43.43
	2111	14.68			
3	1796	12.75			
1.25"	1854	13.98			
Immersion					
	2318	19.12			
	2566	20.85			
	2651	22.75			
	2813	24.67			
	3011	28.02			
	3074	28.64			

HOSKINS MANUFACTURING COMPANY

CABLE ADDRESS "THERMO" 4445 LAWTON AVENUE • DETROIT 8, MICHIGAN

January 8, 1964

Auto-Control Laboratories Inc.
5251 W. Imperial Highway
Los Angeles 45, Calif.

A F F I D A V I T

This is to certify that the 30 double feet of .020" diameter Tungsten versus Tungsten-26% Rhenium thermocouple wires shipped Jan. 8, 1964 against your Order 17768 has the following temperature/emf characteristics:

Tungsten Lot # 5
Tungsten-26% Lot # 2603

Temperature (°F)	EMF (Mv.)	Temperature (°F)	EMF (Mv.)
200	.287	2200	18.537
400	1.049	2400	20.875
600	2.159	2600	23.112
800	3.615	2800	25.441
1000	5.429	3000	27.580
1200	7.297	3200	29.713
1400	9.424	3400	31.893
1600	11.583	3600	33.805
1800	13.853	3800	35.668
2000	16.199	4000	37.485
		4200	39.185

Any liability under this affidavit is limited by the terms and conditions appearing on our General Order.

HOSKINS MANUFACTURING COMPANY

TABLE II

L. C. Lane

Sales Service Section

ELECTRICAL RESISTANCE, THERMO-ELECTRIC AND SPECIAL PURPOSE Alloys

3.1.3.2 Notes on Oven Operation

The oven was quite easily controllable up to about 4500°F. As power was increased beyond that point, great care must be exercised in increasing temperature. At the end of Run No. 3, a relatively large power increase caused the tungsten cavity to melt and the gauge tip was in turn melted before the power could be cut back.

This same effect was noted in Run No. 4. Power had been interrupted at the 4800°F point after the output was recorded. After power was restored, a fast run-up was made, while tracking the cavity optically and also tracking the output on the L & N Potentiometer. Just after the output reading at 43.43 millivolts was recorded, a slight power increase melted a hole in the side of the cavity. Power was immediately cut off, but on cooling, it was found that the gauge sheath was fused to the inside of the cavity. The probe still had continuity at this point. When an attempt was made to remove the gauge from the cavity, the tip broke.

It is of great interest to note that, even after cycling the gauge several times during the run, there was no evidence of recrystallization. That is, the radial grain structure remained small, which is highly desirable in considering that small grain size is associated with retention of strength at elevated temperatures.

3.1.3.3 Stability of W-W26Re Thermocouples

Little reliable background data is available regarding stability of tungsten-tungsten 26 rhenium thermocouples at and near the temperatures of interest. ACL plans to conduct stability tests in the near future. In preparation for such tests a literature search has disclosed a report*, in which such investigations are discussed.

Figure 2, reproduced from this report, shows the effect of temperature cycling a test thermocouple several times in a hydrogen atmosphere. The highest temperature to which the thermocouple was cycled was 2300°C, which approaches the temperatures of interest

*Stability of Rhenium/Tungsten Thermocouples in Hydrogen, Keuther & Lachman, ISA Journal, March 1960.

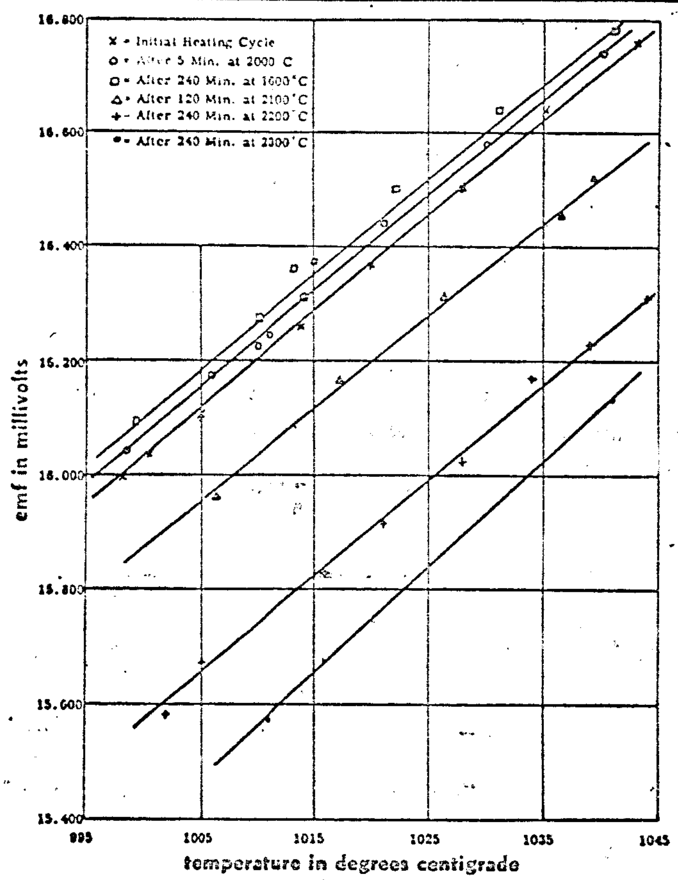

Figure 2. Stability of rhenium/tungsten thermocouples in hydrogen.

FIGURE 2

3.1.3.3 Stability of W-W26Re Thermocouples (Contin.)

in this program. The authors conclude that their tests were of value in that the error of less than 4% at 1000°C lessens to 1.5% at 1500°C and was not detectable at 2000°C. They believe that the instability observed during these tests is due either to changes in chemical or physical properties, or to the addition of impurities by the furnace in which the thermocouples were heated. The tests were run in the presence of tungsten, molybdenum and zirconium, and impurities in the refractories of the oven. ACL agrees with the authors that the tests may be even more significant because of the presence of the contaminants. Results of ACL stability tests will be included in future reports.

SECTION IV

PROGRAM FOR NEXT INTERVAL

Objectives for the Interval 1 March 1964 to 1 April 1964 are:

a. Continue calibrations in the 4000°F to 5400°F range.

b. Continue tests of oxidation resistant coatings.

c. Review design of body and immersed portion of gauges.

SECTION V

STATEMENT OF MAN HOURS

5.0 Hours by Category

Category	Previous Periods	Current Period	To Date
Engineering	694.00	70.5	764.5
Clerical	125.50	10.0	135.5
Fabrication	709.50	54.0	763.5
Consulting	20.50	-0-	20.5
Drafting	61.00	-0-	61.0